



PATENT 1422-0416PUS2

IN THE UNITED STATES PATENT & TRADEMARK OFFICE

In Re Application of :  
Yutaka KANAMARU et al. : Group Art Unit: 1756  
Serial No.: 10/815,790 :  
Filed: April 2, 2004 : Examiner: C. D. RODEE  
For: TONER FOR DEVELOPMENT OF ELECTROSTATIC LATENT  
IMAGES

DECLARATION UNDER 37 C.F.R. 1.132

HONORABLE COMMISSIONER OF  
PATENTS & TRADEMARKS  
WASHINGTON, D.C. 20231

Sir:

I, Yutaka KANAMARU, residing in Wakayama-shi, Wakayama-ken, Japan,  
hereby declares and states as follows:

1. That I am one of the co-inventors of U.S. Application Serial No. 10/815,790 filed on April 2, 2004, entitled TONER FOR DEVELOPMENT OF ELECTROSTATIC LATENT IMAGES. I am thoroughly familiar with the contents of said Application, its prosecution before the United States Patent and Trademark Office and the references cited therein.
2. That I am a graduate of Graduate School of Osaka City University, Faculty of Applied Chemistry in the year 1985, majoring in organic chemistry.
3. That I have been employed in Kao Corporation in the year 1985 and have been assigned to the Research Laboratories.

4. That I have been involved in the research and development of toner since 1987.

5. That the following experiments were conducted by myself or under my direct supervision and control in order to verify that the present invention exhibits superior effects over Uchida et al. (Uchida), USP 4,863,824.

## EXPERIMENTS

### Preparation of Polyester

Resins 2-2c and 2-3 used in Example 5 of Uchida were reproduced by the following methods.

Starting materials the recipes of which are shown in Table A were charged into a four-necked flask (capacity, 2 l) equipped with a thermometer, a stainless steel stirrer, a nitrogen introducing glass pipe and a dropping condenser. The flask was set in a mantle heater and reaction was carried out with stirring at 200°C in a nitrogen atmosphere. The progress of the reaction was monitored by an acid value measurement. At the time when a predetermined acid value was attained, the reaction was quenched and the reaction product was cooled to room temperature to yield polyester samples in the form of a pale yellow solid.

The characteristic values of the polyester samples are noted in Table B.

Table A

Polyester No.	Alcohol component		Acid component	
	Polyoxypropylene (2.2)-2,2-bis (4-hydroxyphenyl) Propane	Trimethylol-propane	Terephthalic Acid	Isododecenyl succinic acid
2-2c	602g (1.75mol)	25.0g (0.19mol)	232g (1.40mol)	172g (0.65mol)
2-3	447g (1.30mol)	67.0g (0.50mol)	232g (1.40mol)	172g (0.65mol)

Table B

Polyester No.	Softening Point (°C)	Glass Transition Point (°C)	Chloroform Insolubles (wt %)
2-2c	143	68	14.3
2-3	102	61	0

#### Preparation of Toner

Polyesters 2-2c and 2-3 with amounts shown in Table C, wax shown in Table C and 10 parts by weight of carbon black "Mogul L" (product of Cabot Corporation) were preliminarily blended and subjected to a standard process consisting of melting, kneading, cooling, grinding and classification. As a result, untreated toners having an average particle size of 10  $\mu$ m were obtained.

The amount 0.3 parts by weight of a hydrophobic silica "H-2000" (manufactured by Wacker Chemical Co.) was blended with 100 parts by weight of each of the resulting untreated toners by using a Henschel mixer to give each of the toners.

Evaluation of Toner

Test Example I

A developer was prepared by blending 32 parts by weight of each of the toners with 768 parts by weight of silicone-coated ferrite carrier (average particle size: 90  $\mu\text{m}$ ).

The same procedures as in Test Example 1 of the present invention were carried out to determine the lowest fixing temperature and the hot-offset generating temperature, except that the copy machine was changed from a modified apparatus of "SF9800" (manufactured by Sharp Corporation) to a modified apparatus of "AR-505" (manufactured by Sharp Corporation), and that the reflective densitometer was changed from RD-915 (manufactured by Macbeth Process Measurements Co.) to "SPM-50" (manufactured by GRETAG). Different apparatuses from those described in the present specification were used in the test because the manufactures and sales of the apparatuses were terminated.

Specifically, each of the developers prepared as described above was loaded on a copy machine [a modified apparatus of "AR-505" (manufactured by Sharp Corporation) which was equipped with an organic photoconductor and a fixing roller having a rotational speed of 250 mm/sec, set to have variable heat roller temperatures, and a fixing unit having no oil applying device]. By sequentially increasing the fixing roller temperature from 90°C to 240°C, the formed images were developed to determine the lowest fixing temperature and the hot offset generating temperature by the following methods. The results are shown in Table C.

## (1) Lowest Fixing Temperature

The lowest fixing temperature used herein referred to the temperature of the fixing roller at which the fixing ratio of the toner exceeded 70%. This fixing ratio of the toner was determined by placing a load of 500 g on a sand-rubber eraser (LION No. 502) having a bottom area of 15 mm × 7.5 mm on a fixed toner image obtained in the fixing device, moving the loaded eraser on the image backward and forward five times, measuring the optical reflective density of the image before or after the eraser treatment with a reflective densitometer "SPM-50" manufactured by GRETAG, and then calculating the fixing ratio by the following equation.

$$\text{Fixing Ratio} = \frac{\text{Optical density after eraser treatment}}{\text{Optical density before eraser treatment}} \times 100$$

## (2) Hot-Offset Generating Temperature

Fixed images were developed at each temperature, and subsequently blank image-transfer paper was conveyed through the fixing roller under the same conditions as above. The "hot offset generating temperature" is referred to a temperature of the fixing roller at which toner dusts were initially generated on the blank paper.

Test Example II

The same procedures as those described in Test Example 2 of the present invention were carried out. Specifically, a 100 ml glass bottle was charged with 10 g of each toner, and the blocking resistance was evaluated after the toners were

allowed to stand under the conditions of 50°C temperature and 26% relative humidity for two weeks in accordance with the following evaluation criteria:

- : Completely no blocking was observed.
- ×: Toner was in a hard caking state.

The results are shown in Table C.

### Test Example III

The same procedures as those described in the portions beginning at page 24, bottom line to page 25, line 8 of the present specification were carried out to determine the durability, except that the copy machine was changed from “SF9800” (manufactured by Sharp Corporation) to “AR-505” (manufactured by Sharp Corporation) for the same reason as Test Example I in the present declaration.

Specifically, the life of the developer was defined as the instance at which the background on the white portion of the printed paper can be visibly confirmed by continuously copying with a copy machine (“AR-505” (manufactured by Sharp Corporation) using an A4 copy sheet having a blackened proportion of 5%, and the number of copies printed until reaching this point is a measure of the durability. Incidentally, the maximum copies were set at 150,000. The results are shown in Table C.

Table C

	Polyester	Amount (parts by weight)	Wax <sup>1)</sup>	Amount (parts by weight)	Melting Point (°C)	Lowest Fixing Temp. (°C)	Hot-Offset Generating Temp. <sup>2)</sup> (°C)	Blocking Resistance	Durability (×1000 sheets)
Toner 1	2-2c 2-3	60 40	---	---	---	128	200	○	150
Toner 2	2-2c 2-3	60 40	Polypropylene Wax	5	135,144	122	240<	○	115
Toner 3	2-2c 2-3	60 40	Carnauba Wax	1	84	102	240<	○	150
Toner 4	2-2c 2-3	60 40	Carnauba Wax	5	84	98	240<	○	150
Toner 5	2-2c 2-3	60 40	Carnauba Wax	7	84	96	240<	×	135
Toner 6	2-2c 2-3	60 40	Rice Wax	5	78	100	240<	○	150
Toner 7	2-2c 2-3	60 40	Fischer- Tropsch Wax	5	72	103	240<	×	100
Toner 8	2-2c 2-3	70 30	Carnauba Wax	5	84	100	240<	○	150
Toner 9	2-2c 2-3	40 60	Carnauba Wax	5	84	96	240<	○	150

1) Polypropylene Wax: "Viscol 660P"

[manufactured by Sanyo Chemical Industries, Ltd., melting points: 135°C, 144°C (2 peaks detected by DSC)]

Carnauba Wax: "Carnauba Wax No. 1" [manufactured by K.K. Kato Yoko, melting point: 84°C]

Rice Wax: "M-90" [manufactured by K.K. Serarika-Noda, melting point: 78°C]

Fischer-Tropsch Wax: "FT-0070" [manufactured by Nippon Seiro Co., Ltd, melting point: 72°C]

2) "240&lt;" means that no offset is generated at 240°C.

## RESULTS AND DISCUSSION

It is found from the above results that Toners 3, 4, 6, 8, and 9 each comprises a given amount of an ester wax having a low melting point, all of which give excellent results in their performances.

On the other hand, Toner 1 not containing an ester wax is unsatisfactory in low-temperature fixing ability and offset resistance, and Toner 2 containing a high-melting point wax, which is the closest example to Toner 5 of Uchida, is unsatisfactory in low-temperature fixing ability and durability.

Toner 5 having an exceedingly high content of the low-melting point wax is unsatisfactory in blocking resistance and durability.

Furthermore, Toner 7 containing a wax other than the ester wax is unsatisfactory in blocking resistance and durability even though the wax was a low-melting point wax. The reason therefor is presumably due to the lowering of the compatibility of the resin binder with the wax, as compared to that with the ester wax.

Therefore, it is clear that by the adjustment of the melting point of the wax but also the kind and the content of the wax, the obtainment of the excellent results for all of low-temperature fixing ability, offset resistance, the blocking resistance and the durability is a surprising effect which would not have been expected by one of ordinary skill in the art simply from the disclosure of Uchida.



6. The undersigned petitioner declares further that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under Section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

7. Further declarant saith not.

  
Yutaka KANAMARU

November 18, 2004

Date